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3,3′-Dimethyl-1,1′-ethylenediimidazolium dibromide

Yu Chen, Wei Song, Juan Xu, Rui-bo Cui and Dan-bi Tian*

Department of Applied Chemistry, College of Science, Nanjing University of Technology, Nanjing 210009, People's Republic of China
Correspondence e-mail: danbi@njut.edu.cn

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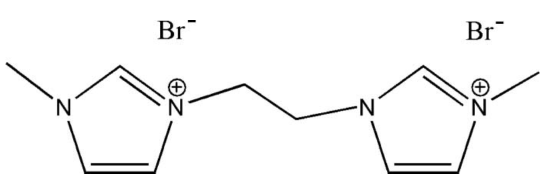
Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.012 Å; R factor = 0.059; wR factor = 0.160; data-to-parameter ratio = 16.6.

The title compound, $C_{10}H_{16}Br_2N_4$, was synthesized by the reaction of 1-methylimidazole and 1,2-dibromoethane in toluene. The complete dication is generated by a crystallographic inversion centre situated at the mid-point of the ethane C—C bond. In the crystal structure, weak intermolecular C—H···Br interactions link the molecules into chains along the b axis and an intramolecular C—H···Br close contact is also present.

Related literature

For general background, see: Ding *et al.* (2007). For related literature, see: Peveling (2001); Takao & Kazuhiko (1997). For bond-length data, see: Allen *et al.* (1987).



Experimental

Crystal data

$C_{10}H_{16}N_4^{2+}\cdot 2Br^-$
M_r = 352.07
Monoclinic, $P2_1/c$
a = 8.4750 (17) Å
b = 8.9620 (18) Å
c = 9.2390 (18) Å
β = 107.73 (3)°
V = 668.4 (3) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 6.05 mm^{-1}
T = 293 K
0.30 × 0.20 × 0.10 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
Absorption correction: ψ scan (North *et al.*, 1968)
T_{min} = 0.264, T_{max} = 0.583
1296 measured reflections
1212 independent reflections
862 reflections with $I > 2\sigma(I)$
R_{int} = 0.021
3 standard reflections every 200 reflections intensity decay: 1%

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.059
$wR(F^2)$ = 0.160
S = 1.01
1212 reflections
73 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.80 e Å^{-3}
$\Delta\rho_{min}$ = −0.85 e Å^{-3}

Table 1
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
C2—H2A···Br	0.93	2.92	3.591 (8)	130
C1—H1B···Br^{i}	0.96	2.97	3.738 (8)	138

Symmetry code: (i) $-x+2, -y+1, -z+1$.

Data collection: *CAD-4 EXPRESS* (Enraf–Nonius, 1985); cell refinement: *CAD-4 EXPRESS*; data reduction: *XCAD4* (Harms & Wocadlo,1995); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *SHELXTL*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: AT2872).

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supporting information

Acta Cryst. (2009). E**65**, o2454   [doi:10.1107/S1600536809036009]

3,3′-Dimethyl-1,1′-ethylenediimidazolium dibromide

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S1. Comment

The title compound is a kind of ionic liquids to be used as green alternatives to volatile organic solvents inelectrochemical, synthetic and separation processes. For general background, see: (Ding *et al.*, 2007). We herein report the crystal structure of the title compound (I).

In the molecule of (I), (Fig. 1), the bond lengths (Allen *et al.*, 1987) and angles are within normal ranges. The whole molecule has an inversion symmetry located on the ethane group of the main molecule.

In the crystal structure, weak intermolecular C—H···Br interactions (Table 1) link the molecules into chains along the *b* axis (Fig.2), in which they may be effective in the stabilization of the structure.

S2. Experimental

The ionic liquid compound was prepared following modified literature procedures (Ding *et al.*, 2007). 1-Methylimidazole (8.21 g, 0.1 mol) was mixed with 1,2-dibromoethane (9.38 g, 0.05 mol) in 100 ml of toluene and refluxed for 24 h; the mixture was cooled to room temperature and filtered. The solids were washed several times with ethyl acetate (800 ml) and the white product dried in vacuum (yield:7.3 g, 54.2%). The product was dissolved in the chloroform and the crystals were obtained by evaporating the chloroform slowly at room temperature for about 9 d.

S3. Refinement

Carbon-bound H atoms were positioned with idealized geometry [aromatic C—H = 0.93 Å, methylene C—H = 0.97 Å and methyl C—H = 0.96 Å] and refined with fixed isotropic displacement parameters [U_{iso}(H) = 1.5U_{eq}(*H*)(methyl C) and U_{iso}(H) = 1.2U_{eq} (aromatic and methylene C)] using a riding model.

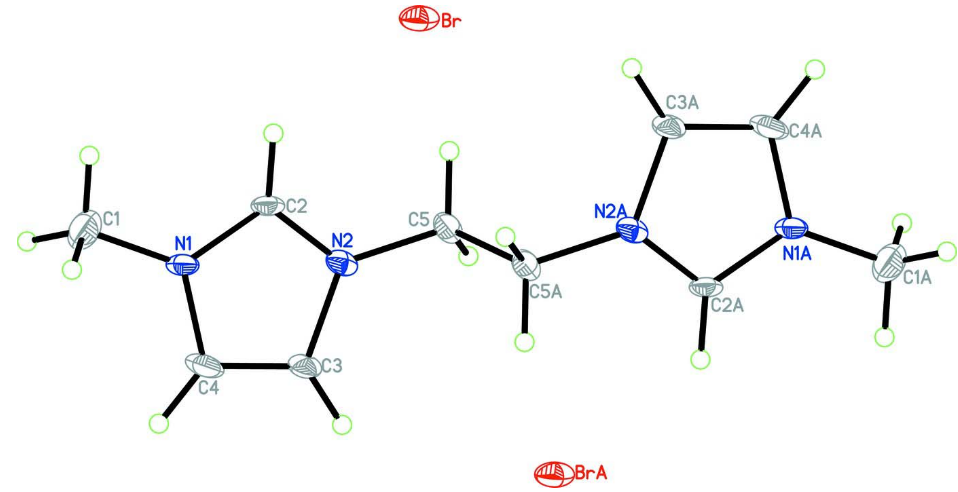

Figure 1

A drawing of the title molecular structure, with the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level. Atoms labeled with the suffixes A are generated by the symmetry operation (-x+1, -y+1, -z+1). Hydrogen bonds are shown by dashed lines.

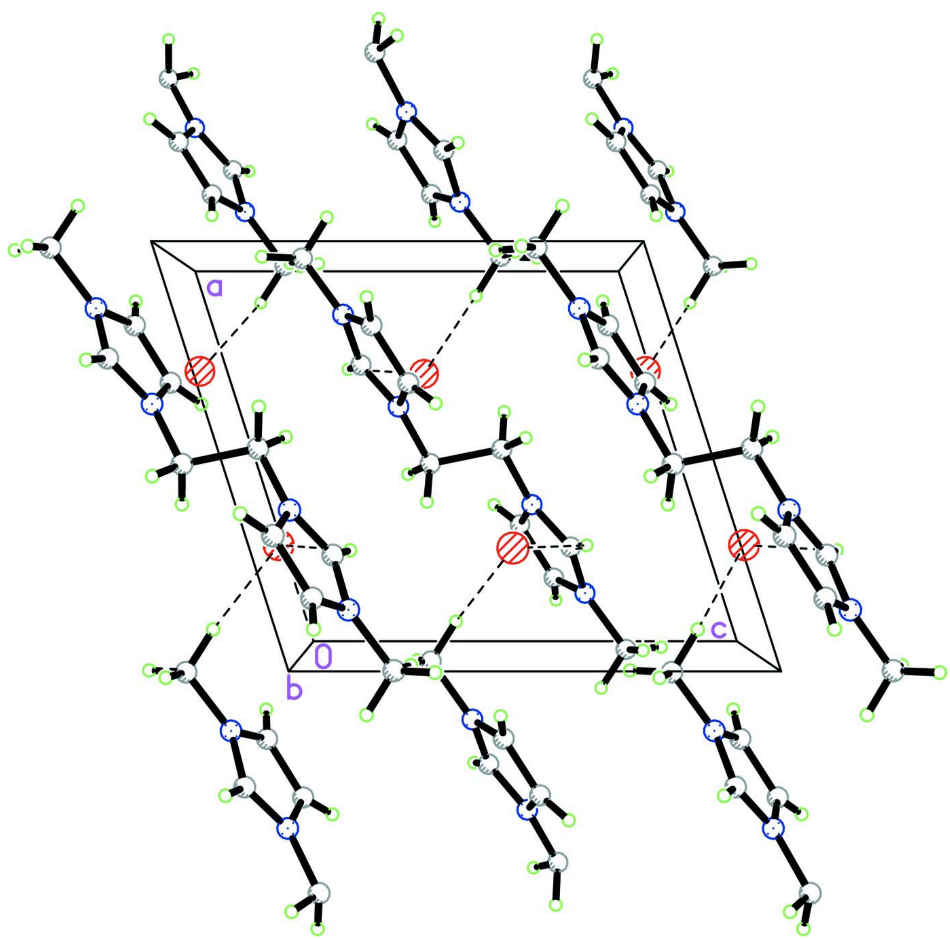

Figure 2

A packing diagram for (I).

3,3′-Dimethyl-1,1′-ethylenediimidazolium dibromide

Crystal data

$C_{10}H_{16}N_4^{2+}\cdot 2Br^-$
$M_r = 352.07$
Monoclinic, $P2_1/c$
Hall symbol: -P 2ybc
$a = 8.4750$ (17) Å
$b = 8.9620$ (18) Å
$c = 9.2390$ (18) Å
$\beta = 107.73$ (3)°
$V = 668.4$ (3) Å^3
$Z = 2$
$F(000) = 348$
$D_x = 1.749$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 25 reflections
$\theta = 9$–13°
$\mu = 6.05$ mm^{-1}
$T = 293$ K
Square, white
0.30 × 0.20 × 0.10 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
$\omega/2\theta$ scans
Absorption correction: ψ scan (North *et al.*, 1968)
$T_{min} = 0.264$, $T_{max} = 0.583$
1296 measured reflections
1212 independent reflections
862 reflections with $I > 2\sigma(I)$
$R_{int} = 0.021$
$\theta_{max} = 25.3°$, $\theta_{min} = 2.5°$
$h = 0\rightarrow10$
$k = 0\rightarrow10$
$l = -11\rightarrow10$
3 standard reflections every 200 reflections
intensity decay: 1%

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.059$
$wR(F^2) = 0.160$
$S = 1.01$
1212 reflections
73 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.1P)^2 + 0.7P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.80$ e Å^{-3}
$\Delta\rho_{min} = -0.85$ e Å^{-3}

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted R-factor wR and goodness of fit S are based on F^2, conventional R-factors R are based on F, with F set to zero for negative F^2. The threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating R-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. R-factors based on F^2 are statistically about twice as large as those based on F, and R- factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	y	z	U_{iso}*/U_{eq}
Br	0.71826 (11)	0.14503 (9)	0.46360 (9)	0.0320 (3)

N1	0.8500 (8)	0.6154 (7)	0.3322 (7)	0.0266 (15)
C1	0.9914 (11)	0.5837 (12)	0.2817 (11)	0.048 (3)
H1A	0.9870	0.4818	0.2488	0.072*
H1B	1.0912	0.5997	0.3639	0.072*
H1C	0.9904	0.6486	0.1987	0.072*
N2	0.6231 (8)	0.5865 (7)	0.3878 (7)	0.0224 (14)
C2	0.7287 (9)	0.5215 (9)	0.3336 (8)	0.0235 (17)
H2A	0.7213	0.4231	0.3003	0.028*
C3	0.6774 (10)	0.7310 (9)	0.4270 (9)	0.0276 (19)
H3A	0.6270	0.8023	0.4713	0.033*
C4	0.8169 (11)	0.7482 (9)	0.3886 (10)	0.035 (2)
H4A	0.8796	0.8349	0.3987	0.042*
C5	0.4817 (10)	0.5171 (10)	0.4162 (9)	0.0265 (18)
H5A	0.4544	0.4255	0.3581	0.032*
H5B	0.3869	0.5834	0.3836	0.032*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Br	0.0506 (5)	0.0167 (5)	0.0261 (5)	0.0002 (4)	0.0081 (4)	−0.0024 (4)
N1	0.039 (4)	0.018 (4)	0.024 (3)	−0.007 (3)	0.011 (3)	−0.008 (3)
C1	0.044 (5)	0.068 (7)	0.039 (6)	−0.016 (5)	0.023 (5)	−0.021 (6)
N2	0.034 (4)	0.015 (3)	0.015 (3)	−0.004 (3)	0.003 (3)	0.004 (3)
C2	0.033 (4)	0.017 (4)	0.018 (4)	−0.005 (3)	0.004 (3)	−0.008 (3)
C3	0.045 (5)	0.011 (4)	0.028 (5)	−0.003 (3)	0.013 (4)	0.000 (3)
C4	0.046 (5)	0.018 (4)	0.040 (5)	−0.012 (4)	0.011 (4)	−0.009 (4)
C5	0.029 (4)	0.026 (4)	0.024 (4)	−0.010 (3)	0.007 (3)	0.001 (4)

Geometric parameters (Å, º)

N1—C2	1.331 (10)	N2—C5	1.443 (9)
N1—C4	1.363 (10)	C2—H2A	0.9300
N1—C1	1.441 (10)	C3—C4	1.343 (12)
C1—H1A	0.9600	C3—H3A	0.9300
C1—H1B	0.9600	C4—H4A	0.9300
C1—H1C	0.9600	C5—C5^{i}	1.514 (15)
N2—C2	1.290 (10)	C5—H5A	0.9700
N2—C3	1.386 (10)	C5—H5B	0.9700
C2—N1—C4	107.4 (7)	N1—C2—H2A	124.8
C2—N1—C1	126.9 (7)	C4—C3—N2	106.7 (7)
C4—N1—C1	125.8 (7)	C4—C3—H3A	126.7
N1—C1—H1A	109.5	N2—C3—H3A	126.7
N1—C1—H1B	109.5	C3—C4—N1	107.5 (7)
H1A—C1—H1B	109.5	C3—C4—H4A	126.2
N1—C1—H1C	109.5	N1—C4—H4A	126.2
H1A—C1—H1C	109.5	N2—C5—C5^{i}	110.5 (8)
H1B—C1—H1C	109.5	N2—C5—H5A	109.6

C2—N2—C3	108.0 (6)	C5^{i}—C5—H5A	109.6
C2—N2—C5	126.1 (7)	N2—C5—H5B	109.6
C3—N2—C5	125.7 (7)	C5^{i}—C5—H5B	109.6
N2—C2—N1	110.3 (7)	H5A—C5—H5B	108.1
N2—C2—H2A	124.8		
C3—N2—C2—N1	−0.9 (8)	N2—C3—C4—N1	−2.0 (9)
C5—N2—C2—N1	−175.6 (7)	C2—N1—C4—C3	1.5 (10)
C4—N1—C2—N2	−0.4 (9)	C1—N1—C4—C3	−178.1 (8)
C1—N1—C2—N2	179.2 (8)	C2—N2—C5—C5^{i}	101.3 (10)
C2—N2—C3—C4	1.8 (9)	C3—N2—C5—C5^{i}	−72.4 (11)
C5—N2—C3—C4	176.5 (7)		

Symmetry code: (i) $-x+1, -y+1, -z+1$.

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C2—H2*A*···Br	0.93	2.92	3.591 (8)	130
C1—H1*B*···Br^{ii}	0.96	2.97	3.738 (8)	138

Symmetry code: (ii) $-x+2, -y+1, -z+1$.